National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 114q

Portland Cement Fineness Standard

This Standard Reference Material (SRM) is intended for use in calibrating fineness testing equipment according to ASTM Standard Methods. The SRM unit consists of 20 glass vials with plastic caps containing powdered cement (each vial is contained in a sealed foil bag). Each vial contains approximately 5 g of cement.

Certified Values and Uncertainties: A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for specific surface area and sieve residue are given in Table 1. The certified values for the surface area are the mean of results from analyses performed by cooperating laboratories. The certified value for sieve residue was calculated from a quadratic fit of NIST data using three sieves having openings ranging from 38 μm to 56 μm.

The expanded uncertainties of the certified values for specific surface area were calculated according to the NIST uncertainty policy described in the NIST Technical Note 1297 [1], and are at the 95 % confidence level. The uncertainties include measurement variability within and between laboratories. The surface area uncertainties also include material variability and the uncertainty of the surface area values for the superseded SRM 114p *Portland Cement Fineness Standard*, which was used as the calibrant for this material. The expanded uncertainty for the sieve residue was computed using a Bayesian analysis and is also at the 95 % probability level. The expanded uncertainty accounts for the variability of random measurement effects, sieve calibrations, and material inhomogeneity.

Table l. Certified Values

Measurand	ASTM Method	Certified Value and Expanded Uncertainty
Specific Surface Area (Blaine)	C 204-96a[(a)]	3818 cm^2/g ± 78 cm^2/g (381.8 m^2/kg ± 7.8 m^2/kg)
Specific Surface Area (Wagner)	C 115-96a[(b)]	2183 cm^2/g ± 160 cm^2/g (218 m^2/kg ± 16 m^2/kg)
Sieve Residue (45 μm residue)	C 430-96[(c)]	0.79 % ± 0.19 %

[(a)] Standard Test Method for Fineness of Portland Cement by Air Permeability Apparatus [Blaine].

[(b)] Standard Test Method for Fineness of Portland Cement by the Turbidimeter [Wagner].

[(c)] Standard Test Method for Fineness of Hydraulic Cement by the 45 μm (No. 325) Sieve.

Expiration of Certification: The certification of SRM 114q is valid, within the measurement uncertainties specified, until **31 December 2016,** provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is contaminated or otherwise modified.

Maintenance of Certification: NIST will monitor representative samples from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration date, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

James St. Pierre, Chief
Materials and Construction Research Division

Gaithersburg, MD 20899
Certificate Issue Date: 24 March 2005
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

The preparation of the material and the coordination of the technical measurements leading to certification were performed by C. Ferraris of the NIST Materials and Construction Research Division.

Statistical consultation on measurement design and analysis of the certification data was performed by W.F. Guthrie and A.I. Avilés of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Stability and Use: This material is considered to be extremely hygroscopic. Based on measurements in 1993 of several earlier renewals of SRM 114, the properties certified are stable as long as the foil bag remains sealed. The specific surface area of cement changes on exposure to the moisture in air. Therefore, this cement should be used immediately after opening the outer foil bag.

Allow the sealed foil bag to equilibrate to testing temperature before opening. To open the pouch, cut off the end with scissors. Fluff the cement in accordance with ASTM Standard C204, Section 3.4, allow the cement to settle for 2 minutes, and then perform the measurement.

Material Selection and Packaging: The desired properties were determined to be generally the same as those selected for the previous issues of SRM 114; however, in order to better represent current cements, the material selected for SRM 114q consists of a finer particle size distribution than previously issued. The Cement and Concrete Reference Laboratory (CCRL) and NIST identified a plant with suitable cement: Lehigh Cement Company (Union Bridge, Maryland)[1] donated 1300 kg of appropriate cement for this SRM. The material selected was Type I according to the ASTM C 150 Standard Classification and had a mass fraction of less than 8 % tricalcium aluminate (C_3A). The material was collected for shipment to NIST directly from the finish mill process stream into bags. Upon arrival at NIST, the cement was blended in a V-blender (1.68 m^3) and then transferred to 208 L (55 gallon) drums lined with 0.015 cm (6 mil) polyethylene liners to minimize hydration of the cement in storage prior to preparation and packaging. Over the next two days, the cement from each drum was sealed in foil bags, each containing about 16 kg of cement. The foil bags were stored in a climate-controlled area. The contents of each bag were subsequently packaged into vials. The vials were then capped and packaged in boxes of about 500 vials per box. The boxes were sequentially labeled from 1 to 118. About 5 boxes were filled per day. Nearly 59 000 glass vials, each containing approximately 5 g of cement, were produced. Each vial was then individually sealed in a foil bag. Vials were selected from the lot by stratified random sampling [2] for both homogeneity and certification analyses. Selected vials were shipped to the participating laboratories for measurements. The remaining vials were packaged into SRM unit boxes of 20 vials each.

Homogeneity Assessment and Certification Analyses: Homogeneity testing of the material was performed on 48 random-selected samples. Measurements of the loss of ignition (LoI) showed no reversible moisture take-up by the cement during packaging. The data received from the round-robin participants were also checked for laboratory-to-laboratory (or day-to-day in the case of sieve residue) variability, box-to-box variability, and vial-to-vial variability. No significant box-to-box or vial-to-vial variability was detected except for the Wagner or sieve residue tests, and therefore it was determined that the samples were homogeneous for the ASTM measurements. Significant vial-to-vial variability was observed using the Blaine test and the certified values reflect this source of uncertainty.

Certification analyses for specific surface areas using ASTM Standard Test Methods C 115-96a and C 204-00 were performed on two samples at each of the participating laboratories. SRM 114p *Portland Cement Fineness Standard* was used for calibration. Raw data were submitted by each laboratory to NIST for tabulation and calculation of surface areas, which for the Blaine test, assumed a density of 3.15 g/cm^3. The density was measured twice at NIST: the results were 3.255 g/cm^3 and 3.248 g/cm^3.

Certification analyses according to ASTM Standard Test Method C 430-96 for the 45 μm sieve residue were performed at NIST on 40 samples from 20 vials of cement.

Laboratories performing certification analyses are members of the CCRL (http://www.ccrl.us) proficiency program. The full list is provided in the report describing the details of the certification process [2].

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Information Values: NIST information values are considered to be of interest to the SRM user, but are not certified because insufficient information is available to assess adequately the uncertainty associated with the values or only a limited number of analyses were performed. Information values for SRM 114q are given in Tables 2 and 3. Table 2 provides the approximate chemical composition determined by ASTM Standard Test Method C114-02. The analysis of this cement (CCRL Portland Cement Proficiency Sample No. 150) was performed by 70 to 170 laboratories; the number of participating laboratories depends on the value measured.

Table 2. Information Values for Chemical Composition

Compound	Mass Fraction (%)	Compound	Mass Fraction (%)
CaO	64.0	K_2O	0.70
SiO_2	20.7	TiO_2	0.30
$A1_2O_3$	4.7	P_2O_5	0.12
Fe_2O_3	3.2	Na_2O	0.07
SO_3	2.4	MgO	2.2
Loss on Ignition	1.67		

Table 3 provides the calculation of cement compounds according to ASTM C 150-02.

Table 3. Information Values for Cement Compounds (Calculation from Table 2)

Compound		Mass Fraction (%)
C_3S	(tricalcium silicate)	60
C_2S	(dicalcium silicate)	14
C_3A	(tricalcium aluminate)	7
C_4AF	(tetracalcium alumino-ferrite)	10

REFERENCES

[1] Taylor, B.N.; Kuyatt, C.E.; NIST Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results* (1993).

[2] Ferraris, C.F.; Avilés A.I.; Guthrie W.; Haupt, R.; *Certification of SRM 114q*; *Phase I*, NIST SP260-161 (2005).

Certificate Revision History: 24 March 2005 (This technical revision corrects the certified values and expanded uncertainties for the measurand); 23 March 2005 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.